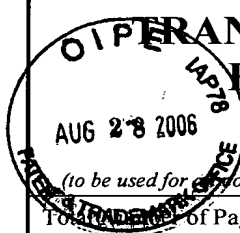
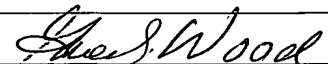
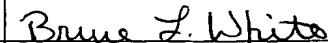


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	<b>TRANSMITTAL FORM</b>		Application Number	10/782,547
			Filing Date	February 19, 2005
			First Named Inventor	Larry F. Rhodes
			Art Unit	1713
			Examiner Name	Robert D. Harlan
Total Number of Pages in This Submission		39	Attorney Docket Number	4262-031383

ENCLOSURES (check all that apply)		
<input checked="" type="checkbox"/> Fee Transmittal Form <input checked="" type="checkbox"/> Fee Attached <input type="checkbox"/> Amendment / Reply <input type="checkbox"/> After Final <input type="checkbox"/> Affidavits/declaration(s)  <input type="checkbox"/> Extension of Time Request <input type="checkbox"/> Express Abandonment Request <input type="checkbox"/> Information Disclosure Statement  <input type="checkbox"/> Certified Copy of Priority Document(s) <input type="checkbox"/> Response to Missing Parts/ Incomplete Application <input type="checkbox"/> Response to Missing Parts Under 37 CFR 1.52 or 1.53	<input type="checkbox"/> Drawing(s) <input type="checkbox"/> Licensing-related Papers <input type="checkbox"/> Petition <input type="checkbox"/> Petition to Convert to a Provisional Application <input type="checkbox"/> Power of Attorney, Revocation Change of Correspondence Address <input type="checkbox"/> Terminal Disclaimer <input type="checkbox"/> Request for Refund <input type="checkbox"/> CD, Number of CD(s) _____ <input type="checkbox"/> Landscape Table on CD	<input type="checkbox"/> After Allowance Communication to TC <input type="checkbox"/> Appeal Communication to Board of Appeals and Interferences <input checked="" type="checkbox"/> Appeal Communication to TC (Appeal Notice, Brief, Reply Brief) <input type="checkbox"/> Proprietary Information <input type="checkbox"/> Status Letter  <input type="checkbox"/> Other Enclosure(s) (please identify below): Return Receipt Post Card Organic Chemistry Excerpt (8 p.) Check in the amount of \$500.00
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SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT			
Firm Name	The Webb Law Firm		
Signature			
Printed Name	Gwen R. Wood, Ph.D.		
Date	August 23, 2006	Reg. No.	51,027

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<p>Transmittal to the Consolidated Appropriations Act, 2005 (H.R. 4818).</p> <p><b>FEE TRANSMITTAL</b></p> <p><b>For FY 2006</b></p> <p>Applicant claims small entity status. See 37 CFR 1.27</p>		<b>Complete if Known</b>	
		Application Number	10/782,547
		Filing Date	February 19, 2005
		First Named Inventor	Larry F. Rhodes
		Examiner Name	Robert d. Harlan
		Art Unit	1713
<b>TOTAL AMOUNT OF PAYMENT</b>		<b>( \$ ) 500.00</b>	
		Attorney Docket No.	4262-031383

**METHOD OF PAYMENT** (check all that apply)

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**FEE CALCULATION** (All the fees below are due upon filing or may be subject to a surcharge.)

**1. BASIC FILING, SEARCH, AND EXAMINATION FEES**

Application Type	FILING FEES		SEARCH FEES		EXAMINATION FEES		Fees Paid (\$)
	Small Entity	Fee (\$)	Small Entity	Fee (\$)	Small Entity	Fee (\$)	
Utility	300	150	500	250	200	100	
Design	200	100	100	50	130	65	
Plant	200	100	300	150	160	80	
Reissue	300	150	500	250	600	300	
Provisional	200	100	0	0	0	0	

**2. EXCESS CLAIM FEES**

Fee Description	Small Entity	Fee (\$)	Fee (\$)
Each claim over 20 (including Reissues)	50	25	
Each independent claim over 3 (including Reissues)	200	100	
Multiple dependent claims	360	180	
<b>Total Claims</b>	<b>Extra Claims</b>	<b>Fee (\$)</b>	<b>Fee Paid (\$)</b>
- 20 or HP = _____ x _____ = _____			
HP = highest number of total claims paid for, if greater than 20.			
<b>Indep. Claims</b>	<b>Extra Claims</b>	<b>Fee (\$)</b>	<b>Fee Paid (\$)</b>
- 3 or HP = _____ x _____ = _____			
HP = highest number of independent claims paid for, if greater than 3.			

**3. APPLICATION SIZE FEE**

If the specification and drawings exceed 100 sheets of paper (excluding electronically filed sequence or computer listings under 37 CFR 1.52(e)), the application size fee due is \$250 (\$125 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).

Total Sheets	Extra Sheets	Number of each additional 50 or fraction thereof	Fee (\$)	Fee Paid (\$)
- 100 = _____ / 50 = _____ (round up to a whole number) x _____ = _____				

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Non-English Specification, \$130 fee (no small entity discount)

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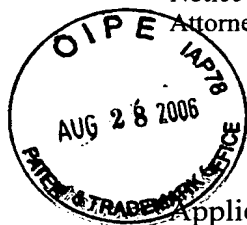
<b>SUBMITTED BY</b>			
Signature		Registration No. 51,027 (Attorney/Agent)	Telephone 412-471-8815
Name (Print/Type)	Gwen R. Wood, Ph.D.	Date	August 23, 2006

This collection of information is required by 37 CFR 1.136. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 30 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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AP  
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Appellants' Brief Under 37 C.F.R. § 41.67  
Application No. 10/782,547  
Paper dated August 23, 2006  
Paper in furtherance of  
Notice of Appeal filed June 8, 2006  
Attorney Docket No. 4262-031383



**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE  
BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES**

Application No. : 10/782,547 Confirmation No. 3252  
Appellants : Larry F. Rhodes et al.  
Filed : February 19, 2005  
Title : VINYL ADDITION POLYCYCLIC OLEFIN POLYMERS  
PREPARED WITH NON-OLEFINIC CHAIN TRANSFER  
AGENTS AND USES THEREOF  
Group Art Unit : 1713  
Examiner : Robert D. Harlan

**MAIL STOP APPEAL BRIEF – PATENTS**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**ON APPEAL FROM THE PRIMARY EXAMINER TO THE  
BOARD OF PATENT APPEALS AND INTERFERENCES**

**APPELLANTS' BRIEF UNDER 37 C.F.R. § 41.67**

Sir:

The present Appeal Brief is submitted in support of the Notice of Appeal filed June 8, 2006, and received June 13, 2006 by the United States Patent and Trademark Office.

I hereby certify that this correspondence is being deposited with the United States Postal Service as First Class Mail in an envelope addressed to Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on August 23, 2006.	
Bruce L. White (Name of Person Mailing Paper or Fee)	08/28/2006 TBESHAH1 00000066 10782547 01 FC:1402
Bruce L. White Signature	08/23/2006 Date

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**I**

**REAL PARTY IN INTEREST**

The real party in interest for the application in this Appeal is assignee Promerus LLC, by virtue of the Assignment dated July 16, 2004, recorded at Reel/Frame 15572/0715 on July 19, 2004 in the United States Patent and Trademark Office.

**Appellants' Brief Under 37 C.F.R. § 41.67**  
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Attorney Docket No. 4262-031383

## **II**

### **RELATED APPEALS AND INTERFERENCES**

As the legal representative of Appellants, the undersigned attorney has no knowledge of any appeals or interferences directly related to this Appeal.

### **III**

#### **STATUS OF CLAIMS**

Claims 1-41, 61, 63, 65 and 67 of this patent application are pending, with claims 1 and 37 being in independent form. Claims 42-60, 62, 64, 66 and 68-75 were canceled pursuant to a restriction requirement. Appellants point out that claim 41 incorrectly was included by the Examiner in Group I, the elected group of claims.

Claims 1-41, 61, 63, 65 and 67 were finally rejected under 35 U.S.C. § 102(a) in the Office Action mailed February 13, 2006 ("the Final Office Action").

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Attorney Docket No. 4262-031383

#### **IV**

#### **STATUS OF AMENDMENTS**

Claim 1 was amended in the Amendment After Final Rejection dated April 10, 2006. According to the Advisory Action of May 8, 2006, the Amendment was entered for the purpose of appeal. A copy of the claims involved in this Appeal is contained in the Appendix attached hereto.



V

**SUMMARY OF CLAIMED SUBJECT MATTER**

In one embodiment set forth in independent claim 1, Appellants' invention is directed to a method of polymerizing poly(cyclic)olefin monomers comprising the steps of:

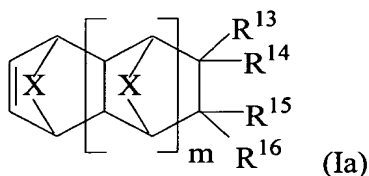
(a) combining a monomer composition comprising one or more poly(cyclic)olefin monomers, a non-olefinic chain transfer agent and an optional activator compound in a reaction vessel to form a mixture; and

(b) adding a polymerization catalyst containing Ni and/or Pd ligated by a monodentate ligand, the catalyst causing the mixture to polymerize;

wherein the non-olefinic chain transfer agent includes one or more compounds selected from the group consisting of H<sub>2</sub>, alkylsilanes, alkylalkoxysilanes, alkylgermanes, alkylalkoxygermanes, alkylstannanes, and alkylalkoxystannanes.

See claim 1 and page 3, paragraph 0009 of the specification.

In another embodiment set forth in independent claim 37, Appellants' invention is directed to an unsaturated monomer comprising Formula (Ia):

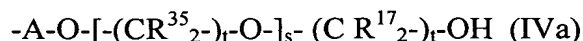


wherein X is selected from -CH<sub>2</sub>-, -CH<sub>2</sub>-CH<sub>2</sub>-, O, S and -NH-; m is an integer from 0 to 5; and each occurrence of R<sup>13</sup>, R<sup>14</sup>, R<sup>15</sup> and R<sup>16</sup> are independently selected from one of the following groups:

(a) H, C<sub>1</sub> to C<sub>25</sub> linear, branched, and cyclic alkyl, aryl, aralkyl, alkaryl, alkenyl and alkynyl;

(b) C<sub>1</sub> to C<sub>25</sub> linear, branched, and cyclic alkyl, aryl, aralkyl, alkaryl, alkenyl and alkynyl containing one or more hetero atoms selected from O, N, and Si;

(c) a hydroxy alkyl ether according to Formula (IVa):



wherein A is a linking group selected from C<sub>1</sub> to C<sub>6</sub> linear, branched, and cyclic alkylene, each occurrence of R<sup>17</sup> is independently selected from H, methyl and ethyl, R<sup>35</sup> is independently selected from H, methyl, ethyl and a halide, t is from 1 to 5, and s is from 0 to 3; and

wherein at least one of R<sup>13</sup>, R<sup>14</sup>, R<sup>15</sup> or R<sup>16</sup> comprise the hydroxyalkyl ether of Formula (IV).

See claim 37 and page 4, paragraph 0011 of the specification.

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Attorney Docket No. 4262-031383

## **VI**

### **ISSUES TO BE REVIEWED ON APPEAL**

1. Whether claims 1-41, 61, 63, 65 and 67 are anticipated under 35 U.S.C. § 102(b) by U.S. Patent No. 6,372,869 to Arthur et al. ("the '869 patent")?

## VII

### ARGUMENT

**A. THE '869 PATENT FAILS TO TEACH OR DISCLOSE EACH AND EVERY ELEMENT OF CLAIMS 1-41, 61, 63, 65 AND 67, AND THEREFORE THE REQUIRED CASE OF ANTICIPATION UNDER 35 U.S.C. § 102(b) OVER THE '869 PATENT HAS NOT BEEN ESTABLISHED.**

**1. The Rejection**

Claims 1-41, 61, 63, 65 and 67 stand rejected under 35 U.S.C. § 102(b) as being anticipated by the '869 patent.

The reasons for rejection are set forth in the Final Office Action. The Final Office Action states that the '869 patent teaches a process for polymerizing cyclic olefins in the presence of a chain transfer agent. Regarding the use of metal catalysts, the Final Office Action states that the '869 patent teaches the use of a Ni metal catalyst formed by contacting monodentate ligands with Ni.

Applicants pointed out in the Response, dated April 10, 2006, that the '869 patent is directed only to bidentate ligands and neither teaches nor suggests the use of Ni and/or Pd catalysts bound to monodentate ligands or using monodentate ligands of a metal as a polymerization catalyst. This argument was characterized as not persuasive in the Final Office Action.

**2. The Prior Art**

The '869 patent discloses bidentate ligands in a process for the polymerization of a polymerizable olefin using as a polymerization catalyst a complex of a bidentate ligand of a metal selected from nickel, iron or cobalt. The process includes using as a chain transfer agent an effective amount of hydrogen, CBr<sub>4</sub> or a compound of the formula R<sup>1</sup>R<sup>2</sup>R<sup>3</sup>SiH, wherein R<sup>1</sup> is an alkyl containing 2 or more carbon atoms, R<sup>2</sup> is an alkyl, and R is hydrogen or an alkyl (column 1, lines 48-56).

In particular, the '869 patent discloses a process for the polymerization of one or more polymerizable olefins, comprising contacting one or more polymerizable olefins, an effective amount of a chain transfer agent selected from the group consisting of hydrogen, CBr<sub>4</sub> and a

compound of the formula  $R^1R^2R^3SiH$ , wherein  $R^1$  is an alkyl containing 2 or more carbon atoms,  $R^2$  is an alkyl, and  $R^3$  is hydrogen or alkyl and an active polymerization catalyst which contains a nickel complex of a ligand of formulae IV through XXXVII (column 1, line 57 to column 5, line 57). The listing of these thirty-three structures immediately following the above-cited disclosures of the '869 patent therefore must be bidentate, not monodentate, ligands.

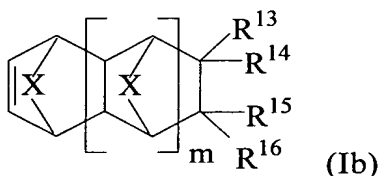
3. **The Required Case of Anticipation Under  
35 U.S.C. § 102(b) Has Not Been Established**

Anticipation under 35 U.S.C. §102 “requires the presence in a single prior art disclosure of all elements of a claimed invention arranged as in the claim...a disclosure that ‘almost’ meets that standard does not ‘anticipate’.” Connell v. Sears, Roebuck & Co., 722 F.2d 1542, 1548, 220 USPQ 193, 198 (Fed. Cir. 1983). In other words, for anticipation under 35 U.S.C. §102 to be found, each and every element set forth in the claim must be found, either expressly or inherently described, in a single prior art reference. (*see*, MPEP §2131 citing to Verdegaal Bros. v. Union Oil Co. of California, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987)). Thus, any feature not directly or explicitly taught must be inherently present. MPEP §706.02. With respect to chemical cases, a prior art reference must clearly and unequivocally disclose the claimed compound or direct those skilled in the art to the compound without any need for picking, choosing, and combining various disclosures. In re Arkley, 455 F.2d 586, 587, 172 U.S.P.Q. 524, 526 (CCPA 1972).

The present invention is directed to a method of polymerizing poly(cyclic)olefin monomers comprising (a) combining a monomer composition comprising one or more poly(cyclic)olefin monomers, a non-olefinic chain transfer agent and an optional activator compound in a reaction vessel to form a mixture; and (b) adding a polymerization catalyst containing Ni and/or Pd ligated by a monodentate ligand, the catalyst causing the mixture to polymerize; wherein the non-olefinic chain transfer agent includes one or more compounds selected from the group consisting of  $H_2$ , alkylsilanes, alkylalkoxysilanes, alkylgermanes, alkylalkoxygermanes, alkylstannanes, and alkylalkoxystannanes.

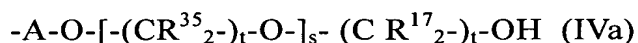
The present invention also is directed to poly(cyclic)olefin-containing polymers prepared according to the above-described method.

The present invention further is directed to hydroxyl functional unsaturated monomers described by Formula (Ib):



where X is selected from  $-\text{CH}_2-$ ,  $-\text{CH}_2\text{CH}_2-$ , O, S, and  $-\text{NH}-$ ; m is an integer from 0 to 5; and each occurrence of  $\text{R}^{13}$ ,  $\text{R}^{14}$ ,  $\text{R}^{15}$  and  $\text{R}^{16}$  is independently selected from one of the following groups, with the proviso that at least one of  $\text{R}^{13}$ ,  $\text{R}^{14}$ ,  $\text{R}^{15}$  and  $\text{R}^{16}$  includes the hydroxyalkyl ether of Formula (IVa) shown below:

- (a) H,  $\text{C}_1$  to  $\text{C}_{25}$  linear, branched, and cyclic alkyl, aryl, aralkyl, alkaryl, alkenyl and alkynyl;
- (b)  $\text{C}_1$  to  $\text{C}_{25}$  linear, branched, and cyclic alkyl, aryl, aralkyl, alkaryl, alkenyl and alkynyl containing one or more hetero atoms selected from O, N, and Si; and
- (c) a hydroxy alkyl ether according to Formula (IVa):



where A is a linking group selected from  $\text{C}_1$  to  $\text{C}_6$  linear, branched, or cyclic alkylene; each occurrence of  $\text{R}^{35}$  is independently selected from H, methyl, ethyl and a halide; each occurrence of  $\text{R}^{17}$  is independently selected from H, methyl and ethyl; t is from 1 to 5; and s is from 0 to 3.

At issue in this appeal is whether the '869 patent discloses a polymerization catalyst containing Ni and/or Pd ligated by a monodentate ligand.

It is asserted in the Final Office Action that the '869 patent anticipates claims 1-41, 61, 63, 65 and 67 because the reference discloses a process for polymerizing cyclic olefins in the presence of a chain transfer agent and the use of a Ni metal catalyst formed by contacting a monodentate ligand. However, the Final Office Action fails to identify where the '869 patent teaches the specific limitation of a Ni metal catalyst formed by contacting a monodentate ligand.

Rather, opposed to the alleged teaching of a monodentate ligand, the '869 patent only teaches a process for polymerizing polymerizable olefin using as a polymerization catalyst, a complex of a bidentate ligand (emphasis added).

This single purpose and the teachings of the '869 patent that are directed to such a purpose are unambiguously previewed in the Abstract, which states: "[t]he molecular weight of polyolefins produced using selected late transition metal complexes of **bidentate** ligands may be lowered by carrying out the polymerization in the presence of hydrogen, a selected silane, or CBr<sub>4</sub> (emphasis added).

In rejecting the claims, the Final Office Action contends, at pages 2-3, that the '869 patent "further teaches the use of Ni metal catalyst formed by contacting mono-dentate ligands with Ni. See Arthur, Example XII." (In two telephone conversations with the Examiner conducted with Bernard Berman, Esq., it was pointed out by Mr. Berman, and recognized by the Examiner, that the correct structure the Examiner meant to refer to in the Office Action is formula XI, not formula XII, at column 2 of the '869 patent.) Such a contention is in opposition to the clear, authoritative statements of the '869 patent. For example, the '869 patent states, at col. 1, lines 48-51, that:

This invention concerns, a process for the polymerization of a polymerizable olefin using as a polymerization catalyst a complex of a **bidentate** ligand of a metal selected from the group consisting of nickel, iron, and cobalt, wherein the improvement comprises, using as a chain transfer agent an effective amount of hydrogen, CBr<sub>4</sub> or a compound of the formula R<sup>1</sup>R<sup>2</sup>R<sup>3</sup>SiH, wherein R<sup>1</sup> is alkyl containing 2 or more carbon atoms, R<sup>2</sup> is alkyl, and R is hydrogen or alkyl (emphasis added).

Continuing from the above, the '869 patent further states, ibid., lines 57-67, that:

This invention also concerns a process for the polymerization of one or more polymerizable olefins, comprising, contacting:

- (a) one or more polymerizable olefins;
- (b) an effective amount of a chain transfer agent selected from the group consisting of hydrogen, CBr<sub>4</sub> and a compound of the formula R<sup>1</sup>R<sup>2</sup>R<sup>3</sup>SiH, wherein R<sup>1</sup> is alkyl, R<sup>2</sup> is alkyl, and R<sup>3</sup> is hydrogen or alkyl;
- (c) an active polymerization catalyst which contains a nickel complex of a ligand of the formula...

The '869 patent then immediately proceeds to list thirty-three structures, one being formula XI. The listing of the thirty-three structures immediately following the above-cited

disclosures of the '869 patent must, therefore, be a listing of bidentate, not monodentate ligands, as the entirety of the '869 patent is silent with respect to monodentate ligands.

Based on the above-cited disclosures of the '869 patent, such patent is directed **only to bidentate ligands**, and we must assume, unless the Examiner submits clear evidence to the contrary, that Example XI of Arthur is a bidentate ligand.

Further, such evidence must overcome the showing that it is known to one of ordinary skill in the art that phenol is recognized as a "bidentate nucleophile" (*see*, Organic Chemistry, 4/e by Francis A. Carey, Chapter 24, page 2 of 6; Phenols, 2001, McGraw Hill; a copy of this reference is attached hereto as Exhibit A.) Indeed, the specification is silent with respect to the use of monodentate ligands of a metal as a polymerization catalyst. The Final Office Action, therefore, is incorrect in contending that the '869 patent teaches the use of a Ni metal catalyst formed by contacting monodentate ligands with Ni. Because the '869 patent does not teach the specific limitation recited in claim 1 of using monodentate ligands of a metal as a polymerization catalyst, it cannot be deemed, under 35 U.S.C. § 102(a), to anticipate claim 1. Claims 2-36, 61, 63, 65 and 67 depend either directly or indirectly on claim 1, and thus also are not anticipated by the '869 patent.

Further, claims 28-30 recite that the polymerization catalyst comprises Pd. In contrast, the '869 patent solely discloses a polymerization catalyst of a complex of a bidentate ligand with a metal selected from nickel, iron or cobalt (column 1, lines 49-51). The '869 patent is silent with respect to the use of a Pd metal. Further still, claim 25 recites an alkylsilane chain transfer agent. The '869 patent is silent with respect to alkylsilane as a chain transfer agent. Thus, the '869 patent fails to disclose a second element in claims 25 and 28-30, i.e., alkylsilane chain transfer agents and Pd, and thus cannot be deemed, under 35 U.S.C. § 102(a), to anticipate claims 25 and 28-30.

With respect to claims 37-40, the '869 patent neither teaches nor suggests an unsaturated monomer comprising Formula Ia, as recited in claim 37, and thus cannot be deemed, under 35 U.S.C. § 102(a), to anticipate claim 37. Because claims 38-40 depend either directly or indirectly on claim 37, they also are not anticipated by the '869 patent.

Claim 41 incorrectly was included by the Examiner in Group I, the group of claims elected by Appellants in response to a restriction requirement. Appellants point out that



the '869 neither teaches nor suggests the elements of claim 41, namely, a negative tone photoresist composition comprising a solvent, a photosensitive acid generator, a crosslinking agent containing one or more functional groups and one or more negative tone imaging polymers comprising poly(cyclic)olefin monomers.

As such, a *prima facie* case of anticipation has not been established, in that specific limitations of the claims are not set forth as being present in the '869 patent. Further, as the '869 patent does not teach or suggest the use of Ni and/or Pd catalysts bound to monodentate ligands, claims 1-41, 61, 63, 65 and 67 cannot be obvious in view of the '869 patent.

Based on these remarks, a *prima facie* case in support of the rejection of claims 1-41, 61, 63, 65 and 67 as being anticipated by the '869 patent has not been met. Accordingly, the Section 102 rejection should be withdrawn.

Appellants' Brief Under 37 C.F.R. § 41.67  
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Paper dated August 23, 2006  
Paper in furtherance of  
Notice of Appeal filed June 8, 2006  
Attorney Docket No. 4262-031383

**CONCLUSION**

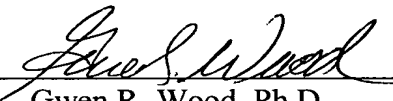
Appellants respectfully submit that all pending claims, claims 1-41, 61, 63, 65 and 67, are patentable and that the present application is in condition for allowance; such action is respectfully requested at an early date.

A check in the amount of \$250.00 is enclosed to cover the Appeal Brief fee.

The Commissioner for Patents is hereby authorized to charge any additional fees which may be required to Deposit Account No. 23-0650. Please refund any overpayment to Deposit Account No. 23-0650. The original and a copy of this Appeal Brief are enclosed.

Respectfully submitted,

THE WEBB LAW FIRM

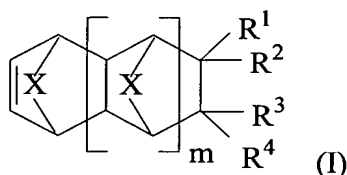
By 

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**CLAIMS APPENDIX**

1. A method of polymerizing poly(cyclic)olefin monomers comprising:
  - (a) combining a monomer composition comprising one or more poly(cyclic)olefin monomers, a non-olefinic chain transfer agent and an optional activator compound in a reaction vessel to form a mixture; and
  - (b) adding a polymerization catalyst containing Ni and/or Pd ligated by a monodentate ligand, the catalyst causing the mixture to polymerize;wherein the non-olefinic chain transfer agent includes one or more compounds selected from the group consisting of H<sub>2</sub>, alkylsilanes, alkylalkoxysilanes, alkylgermanes, alkylalkoxygermanes, alkylstannanes, and alkylalkoxystannanes.
2. The method of claim 1, wherein the non-olefinic chain transfer agent comprises alkylsilanes and/or alkylalkoxysilanes.
3. The method of claim 1, wherein the non-olefinic chain transfer agent comprises H<sub>2</sub>.
4. The method of claim 1, wherein an activator is present and the activator is characterized as having a functional group containing an active hydrogen with a pK<sub>a</sub> of at least about 5.
5. The method of claim 4, wherein the functional group is hydroxyl or carboxylic acid.
6. The method of claim 4, wherein the functional group is -OH.
7. The method of claim 4, wherein the activator is a compound containing an -OH functional group selected from water and C<sub>1</sub>-C<sub>24</sub> linear, branched, and cyclic alkyl, aryl, and alkaryl moieties containing at least one hydroxyl group.

8. The method of claim 1, wherein the poly(cyclic)olefin monomers comprise a first monomer according to Formula (I):



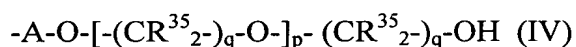
wherein X is selected from  $-\text{CH}_2-$ ,  $-\text{CH}_2\text{CH}_2-$ , O, S, and  $-\text{NH}-$ ; m is an integer from 0 to 5; and each occurrence of  $\text{R}^1$ ,  $\text{R}^2$ ,  $\text{R}^3$  and  $\text{R}^4$  is independently selected from one of the following groups:

- a) H, halogen, linear, branched or cyclic  $\text{C}_1$  to  $\text{C}_{30}$  alkyl, aryl, aralkyl, alkaryl, alkenyl or alkynyl;
- b) linear or branched  $\text{C}_1$  to  $\text{C}_{24}$  halohydrocarbyls,  
 $-(\text{CH}_2)_n\text{C}(\text{O})\text{OR}^*$ ,  $-(\text{CH}_2)_n\text{C}(\text{O})\text{OR}'$ ,  $-(\text{CH}_2)_n\text{OR}$ ,  $-(\text{CH}_2)_n\text{OC}(\text{O})\text{R}$ ,  $-(\text{CH}_2)_n\text{C}(\text{O})\text{R}$ ,  
 $-(\text{CH}_2)_n\text{OC}(\text{O})\text{OR}'$ ,  $-(\text{CH}_2)_n\text{C}(\text{R})_2\text{CH}(\text{R})(\text{C}(\text{O})\text{OR}^{**})$ ,  $-(\text{CH}_2)_n(\text{CR}_2)_n\text{CH}(\text{R})(\text{C}(\text{O})\text{OR}^{**})$ ,  
 $-(\text{CH}_2)_n\text{C}(\text{OR}^{***})(\text{CF}_3)_2$ ,  $-(\text{CR}''_2)_n\text{OR}$ ,  $-\text{CH}_2-[\text{O}(\text{CH}_2)_n]_{m^*}-\text{C}(\text{OR}^{***})(\text{CF}_3)_2$ ,  
 $-(\text{CH}_2)_n\text{C}(\text{R})_2\text{CH}(\text{C}(\text{O})\text{OR}^{**})_2$ ,  $-(\text{CH}_2)_n\text{C}(\text{O})\text{OH}$ ,  $\text{CH}_2)_n\text{C}(\text{R}^*)_2\text{CH}(\text{R}^*)(\text{C}(\text{O})\text{OH})$ ,  
 $-(\text{CH}_2)_n\text{C}(\text{O})\text{O}-\text{R}^{18}$ ,  $-(\text{CH}_2)_n\text{C}(\text{CY}_3)_2\text{OH}$ , and  $-(\text{CH}_2)_n\text{C}(\text{R}^*)_2\text{CH}(\text{C}(\text{O})\text{OH})_2$ ; where each occurrence of R is independently selected from H and linear or branched  $\text{C}_1$  to  $\text{C}_{10}$  alkyl;  $\text{R}'$  is a linear or branched  $\text{C}_1$  to  $\text{C}_{10}$  alkyl or alkylol;  $\text{R}''$  is selected from H and halogen; n and  $m^*$  are each an integer from 0 to 10;  $\text{R}^*$  represents an acid labile group cleavable by a photoacid generator;  $\text{R}^{**}$  is selected from  $\text{R}'$  and  $\text{R}^*$  as defined above and tertiary  $\text{C}_4$  to  $\text{C}_{20}$  alkyl and cycloalkyl,  $\text{C}_1$  to  $\text{C}_6$  trialkylsilyl groups, and  $\text{C}_4$  to  $\text{C}_{20}$  oxoalkyl;  $\text{R}^{***}$  is selected from H,  $-\text{CH}_2\text{OR}'''$ ,  $-\text{C}(\text{O})\text{OR}'''$  and  $-\text{C}(\text{O})\text{R}'''$ , where  $\text{R}'''$  is selected from methyl, ethyl, t-butyl, and  $\text{C}_1$  to  $\text{C}_{20}$  linear or branched cycloaliphatic,  $\text{R}^{18}$  is selected from H, and linear, branched or cyclic

C<sub>1</sub>-C<sub>24</sub> alkyl, aryl, aralkyl, and alkaryl, Y is selected from F and Cl and at least one occurrence of Y is F;

c) C<sub>1</sub> to C<sub>30</sub> linear, branched, or cyclic alkyl, aryl, aralkyl, alkaryl, alkenyl or alkynyl containing one or more hetero atoms selected from O, N, and Si;

d) a hydroxy alkyl ether according to Formula (IV):



wherein A is a linking group selected from C<sub>1</sub> to C<sub>6</sub> linear, branched, or cyclic alkylene, each occurrence of R<sup>35</sup> is independently selected from H, methyl, ethyl and a halide, q is from 1 to 5, and p is from 0 to 3;

e) a group according to Formula (V):



where R<sup>36</sup> is a linear, branched or cyclic C<sub>1</sub> to C<sub>30</sub>, optionally partially or completely halogenated, alkylene, arylene, aralkylene, alkarylene, alkenylene or alkynylene linking group and Z is a functional group selected from hydroxyl, carboxylic acid, amine, thiol, isocyanate and epoxy; and

f) C<sub>r</sub>X<sup>"</sup><sub>2r+1</sub>, wherein X<sup>"</sup> is independently a halogen selected from fluorine, chlorine, bromine or iodine and r is an integer from 1 to 20.

9. The method of claim 8, wherein m=0.

10. The method of claim 8, wherein m=0, R<sup>1</sup> is the hydroxy alkyl ether according to Formula (IV), and R<sup>2</sup>, R<sup>3</sup>, and R<sup>4</sup> are each H in the poly(cyclic)olefin monomer.

11. The method of claim 8, wherein A is methylene or ethylene, each occurrence of R<sup>35</sup> is H, q is from 2 to 5, and p is 0 in the poly(cyclic)olefin monomer.

12. The method of claim 8, wherein  $m=0$ , X is  $-\text{CH}_2-$ ,  $\text{R}^1$ ,  $\text{R}^2$  and  $\text{R}^3$  are hydrogen, and  $\text{R}^4$  is  $-(\text{CH}_2)_n-\text{C}(\text{OR}^{***})-(\text{CF}_3)_2$  where n and  $\text{R}^{***}$  is as defined above.

13. The method of claim 8, wherein  $m=0$ , X is  $-\text{CH}_2-$ ,  $\text{R}^1$  and  $\text{R}^2$  are hydrogen and  $\text{R}^3$  and  $\text{R}^4$  are  $\text{C}_r\text{X}''_{2r+1}$ , wherein  $\text{X}''$  is independently a halogen selected from fluorine, chlorine, bromine or iodine and r is an integer from 1 to 20.

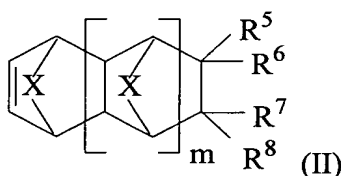
14. The method of claim 1, wherein the poly(cyclic)olefin monomer is selected from the group consisting of  $\alpha,\alpha$ -bis(trifluoromethyl)bicyclo[2.2.1]hept-5-ene-2-ethanol, 5-norbornene-2-methanol hydroxyethylether, t-butylester of norbornene 5-carboxylic acid, hydroxyethylester of 5-norbornene carboxylic acid, trimethylsilane ester of 5-norbornene carboxylic acid, 5-norbornene-2-methanol acetate, 5-norbornene-2-methanol, 5-norbornene-2-ethanol, 5-triethoxysilylnorbornene, 1-methylcyclopentyl ester of 5-norbornene carboxylic acid, tetrahydro-2-oxo-3-furanyl ester of 5-norbornene carboxylic acid, and mixtures thereof.

15. The method of claim 8, wherein the acid labile groups, denoted  $\text{R}^*$ , in the poly(cyclic)olefin monomer are selected from the group consisting of  $-\text{C}(\text{CH}_3)_3$ ,  $-\text{Si}(\text{CH}_3)_3$ , isobornyl, 2-methyl-2-adamantyl, tetrahydrofuranyl, tetrahydropyranyl, 3-oxocyclohexanonyl, mevalonic lactonyl, dicyclopropylmethyl, dimethylcyclopropylmethyl and mixtures thereof.

16. The method of claim 8, wherein  $\text{R}^{**}$  in the poly(cyclic)olefin monomer is selected from the group consisting of tert-butyl, tert-amyl, 1,1-diethylpropyl, 1-methylcyclopentyl, 1-ethylcyclopentyl, 1-butylcyclopentyl, 1-methylcyclohexyl, 1-ethylcyclohexyl,

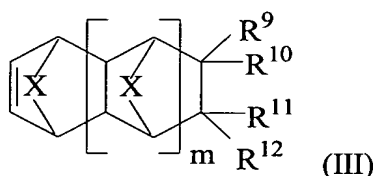
1-butylcyclohexyl, 1-ethyl-2-cyclopentenyl, 1-ethyl-2-cyclohexenyl, 2-ethyl-2-adamantyl, trimethylsilyl, triethylsilyl and dimethyl-tert-butylsilyl, 3-oxocyclohexyl, 4-methyl-2-oxooxan-4-yl, and 5-methyl-2-oxooxolan-5-yl.

17. The method of claim 8, wherein the poly(cyclic)olefin monomers further comprise a second monomer according to Formula (II):



wherein  $m$  is an integer from 0 to 5;  $X$  is selected from  $-\text{CH}_2-$ ,  $-\text{CH}_2\text{CH}_2-$ ,  $\text{O}$ ,  $\text{S}$ , and  $-\text{NH}-$ ;  $\text{R}^5$  to  $\text{R}^8$  are independently selected from  $\text{H}$ ,  $-(\text{CH}_2)_n\text{C}(\text{O})\text{OR}''$ ,  $-(\text{CH}_2)_n\text{OR}'$ ,  $\text{Si}(\text{OR}')_3$ ,  $-(\text{CB}_2)_n\text{OC}(\text{O})\text{R}''$ ,  $-(\text{CH}_2)_n\text{OC}(\text{O})\text{OR}''$ ,  $-(\text{CH}_2)_n\text{C}(\text{O})\text{R}'$ ,  $-(\text{CH}_2)_n\text{C}(\text{R}^*)_2\text{CH}(\text{R}^*)(\text{C}(\text{O})\text{OR}^{**})$ ,  $-(\text{CH}_2)_n\text{C}(\text{R}^*)_2\text{CH}(\text{C}(\text{O})\text{OR}^{**})_2$ ,  $-\text{C}(\text{O})\text{O}-(\text{CH}_2)_n\text{OR}'$  and  $-(\text{CH}_2)_n\text{O}-(\text{CH}_2)_n\text{OR}'$ , wherein  $n$  is independently an integer from 0 to 10;  $\text{B}$  is hydrogen or a halogen;  $\text{R}^*$  is independently hydrogen, a halogen,  $\text{C}_1$  to  $\text{C}_{10}$  linear or branched alkyl or cycloalkyl, or  $\text{C}_1$  to  $\text{C}_{10}$  linear or branched halogenated alkyl or cycloalkyl;  $\text{R}^{**}$  is independently  $\text{C}_1$  to  $\text{C}_{10}$  linear or branched alkyl or cycloalkyl or  $\text{C}_1$  to  $\text{C}_{10}$  linear or branched halogenated alkyl cycloalkyl;  $\text{R}'$  is independently hydrogen, a linear or branched ( $\text{C}_1$  to  $\text{C}_{10}$ ) alkyl group or cycloalkyl group or a linear or branched ( $\text{C}_1$  to  $\text{C}_{10}$ ) halogenated alkyl group or halogenated cycloalkyl group; and  $\text{R}''$  is independently  $\text{C}_1$  to  $\text{C}_{10}$  linear or branched alkyl or halogenated alkyl,  $\text{C}_4$  to  $\text{C}_{20}$  monocyclic or polycyclic cycloaliphatic or halogenated cycloalkyl moiety, a cyclic ether, a cyclic ketone or a cyclic ester (lactone), wherein each of the cyclic ether, ketone and ester can be halogenated or not.

18. The method of claim 17, wherein  $m=0$  in the second monomer.
19. The method of claim 17, wherein the cycloaliphatic groups of  $R^*$  are selected from the group consisting of cyclopropyl, cyclobutyl, cyclopentyl, cyclohexyl, 1-adamantyl, and 1-norbornene.
20. The method of claim 17, wherein the groups  $R^1$  to  $R^4$  and  $R^5$  to  $R^8$  in the poly(cyclic)olefin monomers of Formula (I) and Formula (II) are independently selected such that three or more poly(cyclic)olefin monomers are included in the monomer composition.
21. The method of claim 8, wherein the poly(cyclic)olefin monomers further comprise a third monomer according to Formula (III):



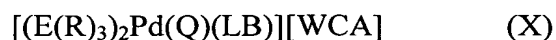
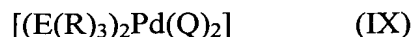
wherein  $m$  is an integer from 0 to 5;  $X$  is  $-\text{CH}_2-$ ,  $-\text{CH}_2\text{-CH}_2-$ ,  $\text{O}$ ,  $\text{S}$ , or  $-\text{NH}-$ ;  $R^9$  to  $R^{12}$  are independently selected from  $\text{H}$ , halogen, linear, branched or cyclic  $\text{C}_1$  to  $\text{C}_{30}$  alkyl, alkylol, aryl, aralkyl, alkaryl, alkenyl or alkynyl; a non-carboxylic acid group containing an active hydrogen with a  $\text{pK}_a$  of 15 or less, and a carboxylic acid substituents selected from the group consisting of those in accordance with the formulas  $-(\text{CH}_2)_q\text{C}(\text{O})\text{OH}$ ,  $(\text{CH}_2)_q\text{C}(\text{R}^{25})_2\text{CH}(\text{R}^{25})\text{C}(\text{O})\text{OH}$  or  $-(\text{CH}_2)_q\text{C}(\text{R}^{25})_2\text{CH}(\text{C}(\text{O})\text{OH})_2$ , wherein  $q$  is an integer from 0 to 10 and each occurrence of  $\text{R}^{25}$  is independently selected from  $\text{H}$ , halogen, linear, branched or cyclic  $\text{C}_1$  to  $\text{C}_{10}$  alkyl, and linear, branched or cyclic  $\text{C}_1$  to  $\text{C}_{10}$  halogenated alkyl; and



wherein at least one of  $R^9$  to  $R^{12}$  is a carboxylic acid substituent as described above.

22. The method of claim 21, wherein  $m=0$  in the third monomer.
23. The method of claim 21, wherein the groups  $R^1$  to  $R^4$  and  $R^9$  to  $R^{12}$  in the poly(cyclic)olefin monomers of Formula (I) and Formula (III) are independently selected such that three or more poly(cyclic)olefin monomers are included in the monomer composition.
24. The method of claim 1, wherein the chain transfer agent is one or more of an alkylsilane or alkylalkoxysilane selected from the group consisting of Si-H containing cyclotetrasiloxanes and compounds according to the formulae  $HSiR^{48}_3$ ,  $HSi(OR^{48})_1R^{48}_2$ ,  $HSi(OR^{48})_2R^{48}_1$ ,  $Si(OSiR^{49})_4$ , and mixtures thereof, wherein each occurrence of  $R^{48}$  is independently selected from linear, branched or cyclic  $C_1$  to  $C_{10}$  alkyl and each occurrence of  $R^{49}$  is independently selected from H and linear, branched or cyclic  $C_1$  to  $C_{10}$  alkyl, where at least one occurrence of  $R^{49}$  is H.
25. The method of claim 24, wherein the alkylsilanes are selected from the group consisting of triethylsilane, tri-isopropylsilane, and mixtures thereof.
26. The method of claim 1, wherein the combination of the monomer composition and catalyst is heated a temperature sufficient to effect polymerization.
27. The method of claim 26, wherein the mixture is heated to a temperature of from 30 to 150°C.
28. The method of claim 1, wherein the polymerization catalyst comprises Pd.

29. The method of claim 1, wherein the polymerization catalyst comprises a catalyst according to one or both of Formulae (IX) and (X):



wherein E is a Group 15 element from the Periodic Table of the Elements, R independently represents hydrogen or an anionic hydrocarbyl containing moiety; Q represents an anionic ligand selected from a carboxylate, thiocarboxylate, and dithiocarboxylate group; LB represents a Lewis base; WCA represents a weakly coordinating anion.

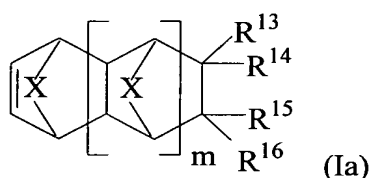
30. The method of claim 1, wherein the polymerization catalyst is selected from the group consisting of *trans*-[Pd(NCMe)(OAc)(P(*I*-propyl)<sub>3</sub>)<sub>2</sub>]B(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>,  
*trans*-[Pd(NCC(CH<sub>3</sub>)<sub>3</sub>)(OAc)(P(*I*-propyl)<sub>3</sub>)<sub>2</sub>]B(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>,  
*trans*-[Pd(OC(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>)(OAc)(P(*I*-propyl)<sub>3</sub>)<sub>2</sub>]B(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>,  
*trans*-[Pd(HOCH(CH<sub>3</sub>)<sub>2</sub>)(OAc)(P(*I*-propyl)<sub>3</sub>)<sub>2</sub>]B(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>,  
*trans*-[Pd(NCMe)(OAc)(P(cyclohexyl)<sub>3</sub>)<sub>2</sub>]B(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>, Pd(OAc)<sub>2</sub>(P(cyclohexyl)<sub>3</sub>)<sub>2</sub>,  
Pd(OAc)<sub>2</sub>(P(*i*-propyl)<sub>3</sub>)<sub>2</sub>, Pd(OAc)<sub>2</sub>(P(*i*-propyl)<sub>2</sub>(phenyl))<sub>2</sub>,  
*trans*-[Pd(NCMe)(OAc)(P(cyclohexyl)<sub>2</sub>(*t*-butyl))<sub>2</sub>]B(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub> and mixtures thereof.

31. The method of claim 1, wherein the polymerization catalyst comprises Ni.

32. The method of claim 1, wherein the molar ratio of monomer to catalyst is from 200:1 to 200,000:1.

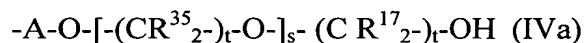
33. A poly(cyclic)olefin polymer prepared according to the method of claim 1.

34. A poly(cyclic)olefin polymer prepared according to the method of claim 8.
35. A poly(cyclic)olefin polymer prepared according to the method of claim 17.
36. A poly(cyclic)olefin polymer prepared according to the method of claim 21.
37. An unsaturated monomer comprising Formula (Ia):



wherein X is selected from -CH<sub>2</sub>-, -CH<sub>2</sub>-CH<sub>2</sub>-, O, S and -NH-; m is an integer from 0 to 5; and each occurrence of R<sup>13</sup>, R<sup>14</sup>, R<sup>15</sup> and R<sup>16</sup> are independently selected from one of the following groups:

- (a) H, C<sub>1</sub> to C<sub>25</sub> linear, branched, and cyclic alkyl, aryl, aralkyl, alkaryl, alkenyl and alkynyl;
- (b) C<sub>1</sub> to C<sub>25</sub> linear, branched, and cyclic alkyl, aryl, aralkyl, alkaryl, alkenyl and alkynyl containing one or more hetero atoms selected from O, N, and Si;
- (c) a hydroxy alkyl ether according to Formula (IVa):



wherein A is a linking group selected from C<sub>1</sub> to C<sub>6</sub> linear, branched, and cyclic alkylene, each occurrence of R<sup>17</sup> is independently selected from H, methyl and ethyl, R<sup>35</sup> is independently selected from H, methyl, ethyl and a halide, t is from 1 to 5, and s is from 0 to 3; and

wherein at least one of R<sup>13</sup>, R<sup>14</sup>, R<sup>15</sup> or R<sup>16</sup> comprise the hydroxyalkyl ether of Formula (IV). IVa ???

38. The monomer of claim 37, wherein  $m=0$ ,  $R^{13}$  is the hydroxy alkyl ether according to Formula (IVa), and  $R^{14}$ ,  $R^{15}$  and  $R^{16}$  are each H.

39. The ethylenically unsaturated monomer of claim 38, wherein A is methylene or ethylene, each occurrence of  $R^{17}$  is H, and s is 0.

40. A polymer comprising repeat units derived from the monomer according to Formula (Ia) of claim 37.

41. A negative tone photoresist composition comprising:

A) a solvent;

B) a photosensitive acid generator;

C) a crosslinking agent containing one or more functional groups; and

D) one or more negative tone imaging polymers comprising the polymers according to claim 34, wherein the polymers contain one or more functional group containing moieties having a functional group that is reactive with the functional groups of the crosslinking agent.

61. The poly(cyclic)olefin polymer of claim 36, wherein the polymer has an optical density of less than 0.2 abs/ $\mu\text{m}$  at an exposure wavelength of 193 nm.

63. The poly(cyclic)olefin polymer of claim 33, wherein the polymer has an optical density of less than 0.2 abs/ $\mu\text{m}$  at an exposure wavelength of 193 nm.

65. The poly(cyclic)olefin polymer of claim 34, wherein the polymer has an optical density of less than 0.2 abs/ $\mu\text{m}$  at an exposure wavelength of 193 nm.
67. The poly(cyclic)olefin polymer of claim 35, wherein the polymer has an optical density of less than 0.2 abs/ $\mu\text{m}$  at an exposure wavelength of 193 nm.

**Appellants' Brief Under 37 C.F.R. § 41.67**  
Application No. 10/782,547  
Paper dated August 23, 2006  
Paper in furtherance of  
Notice of Appeal filed June 8, 2006  
Attorney Docket No. 4262-031383

**EVIDENCE APPENDIX**

Exhibit A: Organic Chemistry, 4/e, FRANCIS A. CAREY, The McGraw-Hill Companies,  
2001, pp. 1-6.

**Appellants' Brief Under 37 C.F.R. § 41.67**  
Application No. 10/782,547  
Paper dated August 23, 2006  
Paper in furtherance of  
Notice of Appeal filed June 8, 2006  
Attorney Docket No. 4262-031383

**RELATED PROCEEDINGS APPENDIX**

None.

# Organic Chemistry, 4/e

by Francis A. Carey

## About the Book

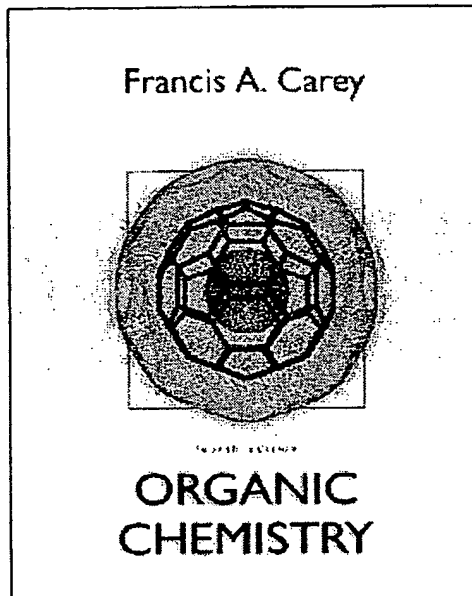
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## ABOUT THE AUTHOR

**Francis A. Carey** is a native of Pennsylvania, educated in the public schools of Philadelphia, at Drexel University (B.S. in chemistry, 1959), and at Penn State (Ph.D. 1963). Following postdoctoral work at Harvard and military service, he joined the chemistry faculty of the University of Virginia in 1966.

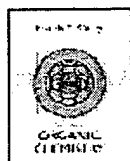
With his students, Professor Carey has published over 40 research papers in synthetic and mechanistic organic chemistry. He is coauthor (with Richard J. Sundberg) of *Advanced Organic Chemistry*, a two-volume treatment designed for graduate students and advanced undergraduates, and (with Robert C. Atkins) of *Organic Chemistry: A Brief Course*, an introductory text for the one-semester organic course.

Since 1993, Professor Carey has been a member of the Committee of Examiners of the Graduate Record

Examination in Chemistry. Not only does he get to participate in writing the Chemistry GRE, but the annual working meetings provide a stimulating environment for sharing ideas about what should (and should not) be taught in college chemistry courses.

Professor Carey's main interest shifted from research to undergraduate education in the early 1980s. He regularly teaches both general chemistry and organic chemistry to classes of over 300 students. He enthusiastically embraces applications of electronic media to chemistry teaching and sees multimedia presentations as the wave of the present.

Frank and his wife Jill, who is a teacher/director of a preschool and a church organist, are the parents of three grown sons and the grandparents of Riyad and Ava.



## Chapter 24: Phenols

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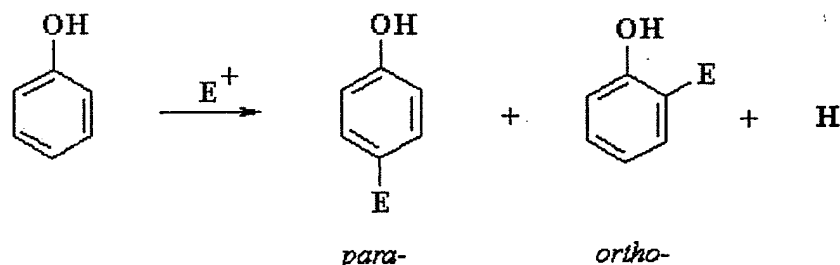


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Reactions of Phenol

Chapter 24: Phenols

### Electrophilic Aromatic Substitution



### Reaction type: Electrophilic Aromatic Substitution

#### Summary

- *Phenols* are potentially very reactive towards electrophilic aromatic substitution.
- This is because the hydroxy group, -OH, is a strongly activating, *ortho- / para-* directing substituent (review)
- Substitution typically occurs *para* to the hydroxyl group unless the *para* position is blocked, then *ortho* substitution occurs.
- The strong activation often means that milder reaction conditions than those used for benzene itself can be used (see table below for a comparison)
- Phenols are so activated that polysubstitution can be a problem (similar problems occur with anilines)

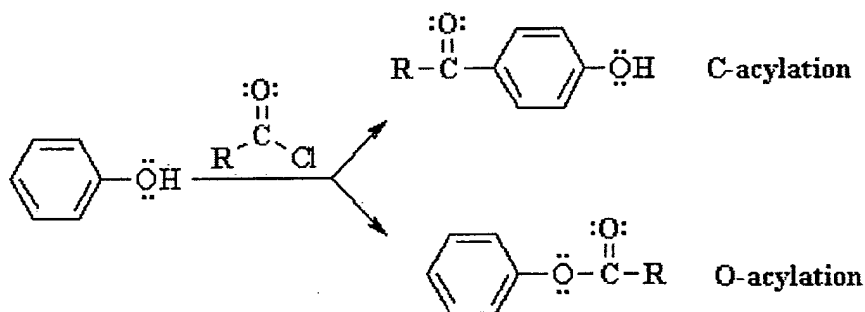
Reaction	Phenol	Benzene
Nitration	dil. $\text{HNO}_3$ in $\text{H}_2\text{O}$ or $\text{CH}_3\text{CO}_2\text{H}$	$\text{HNO}_3 / \text{H}_2\text{SO}_4$
Sulfonation	conc. $\text{H}_2\text{SO}_4$	$\text{H}_2\text{SO}_4$ or $\text{SO}_3 / \text{H}_2\text{SO}_4$
Halogenation	$\text{X}_2$	$\text{X}_2 / \text{Fe}$ or $\text{FeX}_3$

Alkylation	ROH / H <sup>+</sup> or RCl / AlCl <sub>3</sub>	RCl / AlCl <sub>3</sub>
Acylation	RCOCl / AlCl <sub>3</sub>	RCOCl / AlCl <sub>3</sub>
Nitrosation	aq. NaNO <sub>2</sub> / H <sup>+</sup>	

## Related reactions

- Electrophilic Aromatic Substitutions reactions of Benzenes
- Electrophilic Aromatic Substitutions reactions of Aryl Ethers
- Electrophilic Aromatic Substitutions reactions of Aryl Amines

## Acylation of Phenols

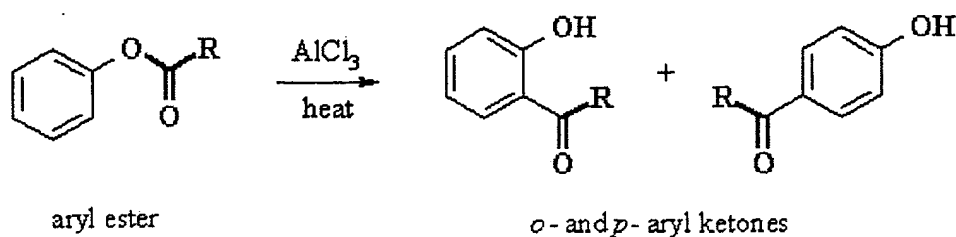


**Reaction type: C-acylation = electrophilic aromatic substitution**

**O-acylation = nucleophilic acyl substitution**

## Summary

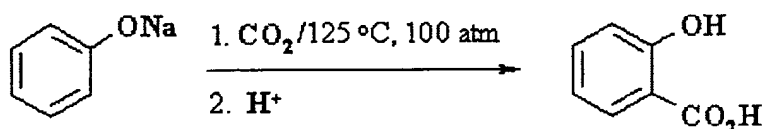
- Phenols are examples of bidentate nucleophiles, meaning that they can react at *two* positions:
  - on the aromatic ring giving an aryl ketone via **C-acylation**, a Friedel-Crafts reaction or,
  - on the phenolic oxygen giving an ester via **O-acylation**, an esterification
- Reagents :
  - C-acylation** : acylating agent (acyl chloride or anhydride) and AlCl<sub>3</sub>
  - O-acylation** : acylating agent (acyl chloride or anhydride)
- The product of **C-acylation** is more stable and predominates under conditions of **thermodynamic control** (i.e. when AlCl<sub>3</sub> is present).
- The product of **O-acylation** forms faster and predominates under conditions of **kinetic control**
- **O-acylation** can be promoted by either:
  - acid catalysis via protonation of the acylating agent, increasing its' electrophilicity or
  - base catalysis via deprotonation of the phenol, increasing its' nucleophilicity.
- It is also known that aryl esters readily rearrange to aryl ketones in the presence of AlCl<sub>3</sub>, a reaction known as the **Fries rearrangement**:



## Related reactions

- Friedel-Crafts Acylation of Benzene
- Conversion of Alcohols to Esters
- Reactions of Acyl Halide

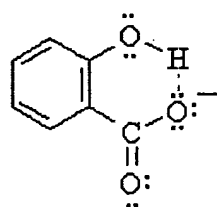
## Carboxylation of Phenols (Kolbe-Schmitt reaction)



## Reaction type: Electrophilic Aromatic Substitution

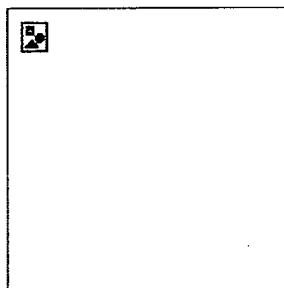
## Summary

- Heating the nucleophilic phenolate salt with carbon dioxide under high pressure / temperature results in regioselective *ortho*-substitution.
- This process is also known as the **Kolbe-Schmitt synthesis**.
- *o*-hydroxybenzoic acid is more commonly known as *salicylic acid*.



The geometry allows for an intramolecular hydrogen bond.

Check out the CHIME image for the intramolecular hydrogen bond by looking at the position of the shared H atom with respect to the two oxygen atoms



## Questions

Aspirin is the acetyl ester of salicylic acid (hence ASA), how would you make aspirin from salicylic acid ?

ANSWER ?

Can you think of other reactions of nucleophilic species with carbon dioxide ? ANSWER ?

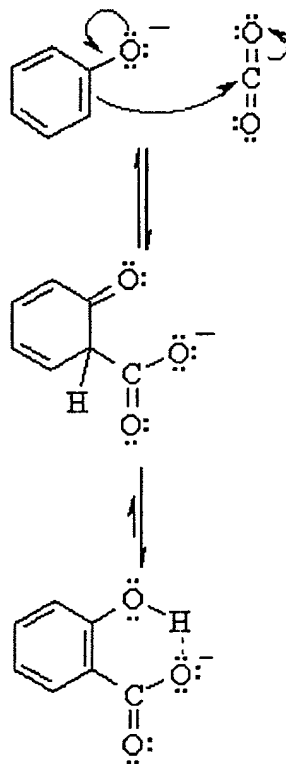
## Study Tip:

Consider the phenolate to be an enolate, hence reactions at the  $\alpha$ -C are typically favored.

### MECHANISM FOR CARBOXYLATION OF PHENOLS

#### Step 1:

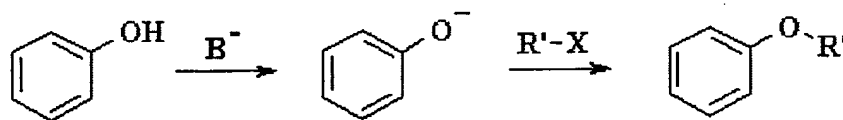
The nucleophilic phenolate (reacting like an enolate) reacts with the electrophilic carbon of carbon dioxide in the *ortho* position (compare this with an Aldol reaction)



#### Step 2:

The non-aromatic cyclohexadienonecarboxylate intermediate tautomerises to the more stable aromatic enol which is further stabilized by an intramolecular hydrogen bond. An acidic work-up will generate the carboxylic acid.

### Preparation of Aryl Ethers



### Reaction type : Nucleophilic Substitution

#### Summary

- Reagents : Use a base such as  $\text{Na}_2\text{CO}_3$  to prepare the phenoxide, then add the alkyl halide.
- Since the reaction is  $\text{S}_{\text{N}}2$ , the halide should be methyl or primary alkyl halides (or tosylates).
- This reaction is the Williamson ether synthesis applied to aromatic alcohols.

**Question :**

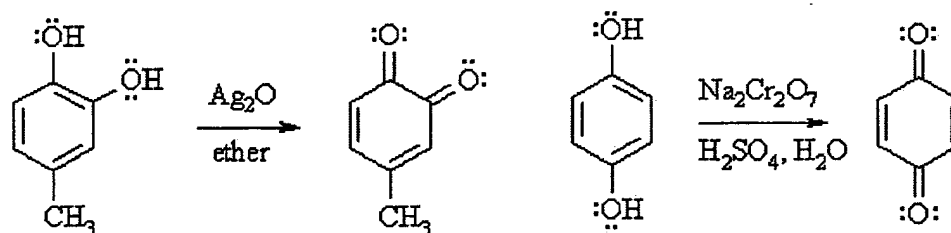
Is this method suitable for the preparation of diaryl ethers such as Ph-O-Ph ? **ANSWER ?**

**Related Reactions**

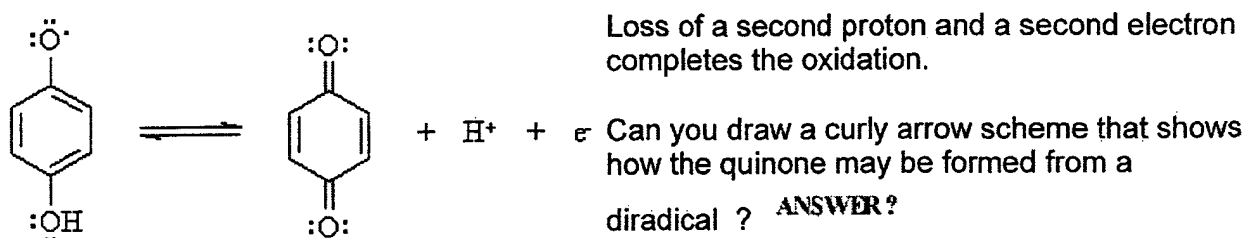
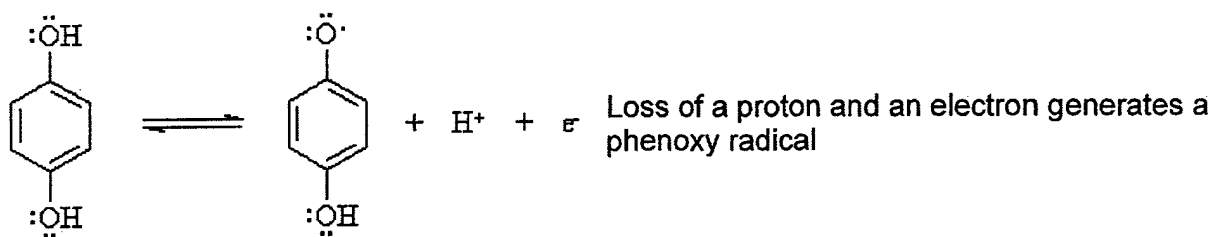
- Williamson Ether Synthesis
- Nucleophilic Substitution reactions of Alkyl Halides

**Oxidation of Phenols****Summary**

- In general, phenols are more easily oxidized than simple alcohols.
- Oxidation can be achieved by reaction with silver oxide ( $\text{Ag}_2\text{O}$ ) or chromic acid ( $\text{Na}_2\text{Cr}_2\text{O}_7$ ), or other oxidizing agents.
- Particularly important are the oxidation of 1,2- and 1,4-benzenediol (pyrocatechol and hydroquinone, respectively) and their derivatives (see examples below):



- These types of systems are important in biological redox-systems such as coenzyme Q and vitamin K.
- Here's a closer look at the two one electron transfers that are believed to take place when hydroquinone is oxidized to benzoquinone

**Related Reactions**

- Oxidations of Alcohols



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